STRUCTURAL TESTING OF INORGANIC BONDED FILAMENT WOUND CYLINDERS

A THESIS
SUBMITTED TO THE DEPARTMENT OF AERONAUTICS AND ASTRONAUTICS
AND THE COMMITTEE ON THE GRADUATE DIVISION
OF STANFORD UNIVERSITY
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF
ENGINEER

By
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LIST OF FIGURES

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I. INTRODUCTION

For some time the Material Science Laboratory of Lockheed Missiles and Space Company, Palo Alto facility, has been producing inorganic bonded filament wound structures. Such structures are known primarily for their unusual insulating properties when filled with epoxy, acrylic, polyethylene, etc. These properties are ablative in nature and have been discussed extensively in various Lockheed reports under the trade name of "Lock-Heat". Structures of this type should not be confused with the fiber reinforced inorganic laminates which have been tested by other organizations. The "Lock-Heat" system differs from others in that it employs plastic rather than inorganic material as the structure matrix.

The inorganic bonded structure would appear to offer some improvement of mechanical properties over the organic bonded structure when in a heat environment of the nature associated with supersonic vehicles. However, prior to this investigation there had been no information available concerning the structural properties of plastic filled inorganic bonded filament wound structures. The possibility that a material could simultaneously serve as heat shielding and primary structure of a vehicle is intriguing indeed, and is the impetus for this investigation.

For this study of the effect on material properties due to inorganic bonding it was decided to test cylinders with three different amounts (percent by weight) of inorganic binder content, keeping all other parameters constant.

Since edge effects on the test results would not be constant for tests with cylinders having various amounts of inorganic binder the well-known NOL (Naval Ordnance Laboratory) ring testing procedures were not used.

The temperature testing technique developed permitted material properties to be determined when the outside surface of the structure was at temperatures in excess of the yield temperature of the matrix material. This more closely approximates the heating and loading environment experienced by supersonic vehicles than the various techniques which require heating prior to loading.

It was not intended to establish rigid design parameters, but rather to demonstrate the trend of some of the structural properties and perhaps outline more specific areas of concern for future studies.

II. TEST SPECIMENS

The cylinders were wound with a constant 26° helix angle on a 36 inch long, 3.000 inch diameter, mandrel which had been wrapped with mylar film. This produced cylinders with a usable length of 30 inches, inside diameter of 3.005 inches and a wall thickness of approximately 1/8 inch. All test samples used for a particular type test and with the same inorganic binder content were cut from the same 30 inch master cylinder, thus minimizing variations in angle of wind and density.

General Electric fused "quartz" QFY - 150 yarn with 1/0 twist was used for the specimens. This yarn is actually amorphous silica ($Si\,O_2$) and consists of two hundred 0.0004 inch diameter monofilament threads per strand. Extensive use is made of such yarn in the "Lock-Heat" process due to its high temperature properties.

The yarn was wound directly from the spindle to the mandrel without any desizing operations. As the yarn was wound, it was saturated with a 5% by weight aqueous solution of colloidal silica ($Si\,O_2$). The porous and fragile master cylinder was allowed to air dry on the mandrel. It was then carefully removed and thoroughly dried at 200° F. Next, the cylinder was fired at 1000°F for approximately 2 hours. During the firing process all of the starch-oil sizing, etc., on the yarn sublimates, providing good clean yarn surfaces for bonding with the matrix material.

The cylinders with higher density inorganic binders were obtained by repeated soaking in silica solution and firing until the desired percent by weight of binder content was obtained. For the current investigation, binder contents of 1%, 10%, and 25% by weight were produced. The cylinders with 1% binder content were produced as control specimens. These cylinders were essentially the same as a standard pure organic system which had been through the identical manufacturing processes as the higher binder content cylinders.

The amount of yarn to be used per master cylinder was to be held constant by using a special weighing system devised by the Material Science Laboratory. After several of the cylinders were wound a small error was found to exist in the weighing system caused by the drag of the yarn tensioning device. The maximum difference in yarn weight of the cylinders was of the order of 3%. This variation was not considered to be significant.

Prior to impregnating with epoxy, the master cylinders were cut to test specimen size. Lengths of 4-1/2 inches were used for the first several tests. It was found from these tests that shorter specimens of 3 inch lengths could be used, thus providing more specimens per master cylinder.

The matrix filler used for the specimens was Shell Epon 815 epoxy with methylnadicanhydrid as the catalyst. The cylinders were first pre-wetted in a low viscous epoxy solution and then vacuum impregnated with the regular epoxy solution. The "B" staging of the epoxy was accomplished by rotating the cylinders on a special rotisserie under infrared lamps. Three thermocouples with their hot junctions in the same cross sectional plane were placed on the outside surface of each cylinder at intervals of 120° and wrapped with a short piece of yarn. These thermocouples then became an integral part of the exterior surface of the cylinders during the "B" staging. The epoxy was cured for 2 hours at 200°F, 2 hours at 250°F, and 6 hours at 300°F.

The ends of the specimens to be used for axial compression testing were potted with epoxy in a teflon die.

III. TEST EQUIPMENT AND INSTRUMENTATION

Each item of collected data was obtained as a continuous plot on one channel of a Sanborn 150 Series six channel recorder. The flexibility of this system in allowing recording channels to be easily switched from DC input (thermocouples) to AC input (strain gage bridges) and vice versa by "plugging in" the necessary preamplifier proved to be a substantial time saver.

A Tinius-Olsen "L" type hydraulic universal testing machine of 120,000 lbs. capacity was used for all load applications. The high range and low range load dials of this machine are actuated by bourdon tube pressure gages and calibrated to read load in pounds. Both of these gages were instrumented with four arm strain gage bridges and wired for input into the Sanborn recorder. This effectively transformed the gages into transducers whose output is proportional to load. Thus, the applied load was displayed as a continuous function on the recorder. An additional feature of this system was that it allowed the loads to be applied at a constant rate by matching the slope of the load curve on the recorder against a series of parallel lines marked on the transparent recorder cover.

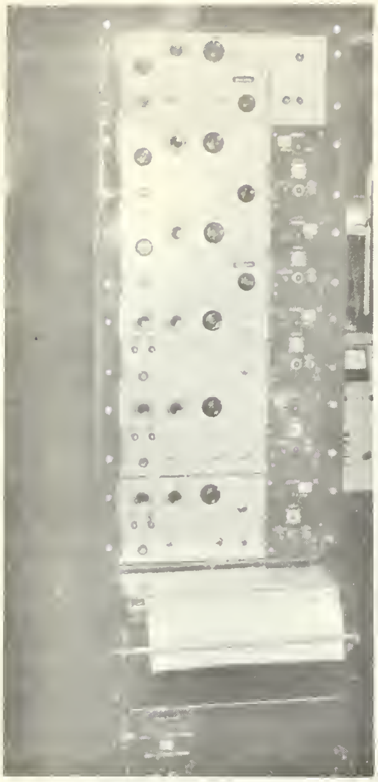
The primary testing equipment is illustrated in Fig. 1 and the testing machine transducers are shown in Fig. 2.

The cross head movement of the testing machine was measured and recorded with a spring beam "positionometer" that determines deflection from the consequent changes in strain of the spring beam. The strain in the spring beam was detected by a four arm strain gage bridge installed on the spring beam and wired for input into the recorder. The "positionometer" and the micrometer arrangement used for calibrating the "positionometer" are illustrated in Fig. 3. It was found that the strain of the spring beam was linear for 1-1/4 inches of "positionometer" deflection.

The heating apparatus consisted of two General Electric quartz 1000 watt heating elements rated at 280 volts, each bent into semicircular shape and together comprising a complete circular heater with an associated supporting device. The supporting device was an aluminum ring with a polished concave reflector on the inside, ventilating ribs on the outside, and three adjustable legs. The circular portion of the quartz heaters fitted into the concave reflector and the projecting ends were supported in spring mounts which allowed for thermal expansion. The two heaters were wired in parallel through a 20 amp. industrial circuit breaker and into a 220 volt power supply. Figure 4 depicts the heating apparatus.

The special ventilating system designed for removing smoke and fumes of the heated epoxy from the laboratory is also illustrated in Fig. 4. Figure 6 shows the installation of the heating and ventilating systems during testing. Smoke moved through ports in the loading head, thence through the crosshead via an attached chimney and was then propelled out the window by compressed air. The compressed air was injected into the system by the circular ring inside the stovepipe elbow shown in Fig. 4. The chimney projected through the crosshead and extended about one inch beyond the circular air inlet. The incoming air created a rapidly moving boundary layer inside the stovepipe which proved very effective in removing smoke.

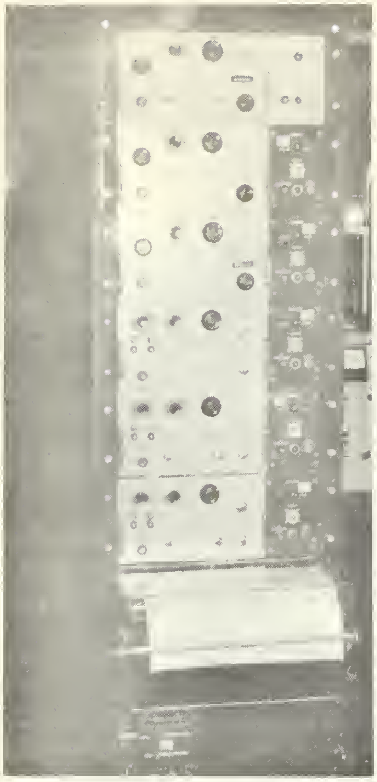
Asbestos covered 30 gage chromel-alumel thermocouple wire was used for all the thermocouples. These thermocouples were made by butt welding with a capacitance discharge welder and were calibrated using a Leeds and Northrup thermocouple checking furnace.



Sanborn Recorder

Tinius-Olsen Testing Machine

FIG. 1. PRIMARY TESTING EQUIPMENT



Sanborn Recorder

Tinius-Olsen Testing Machine

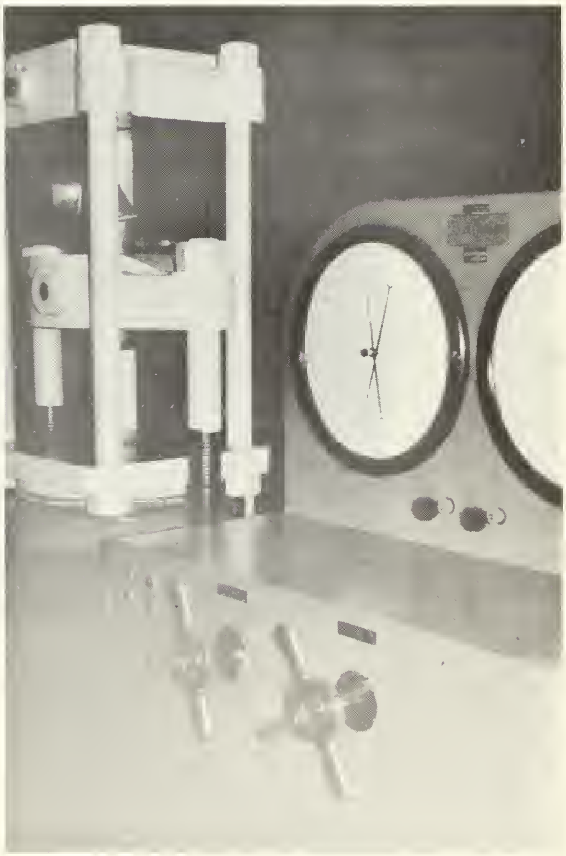
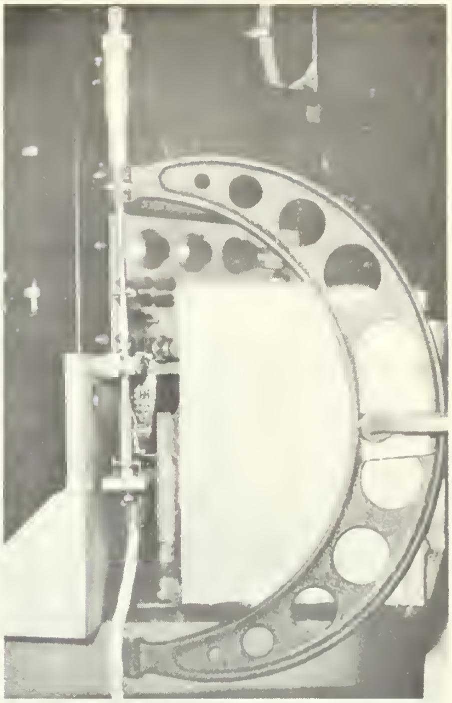
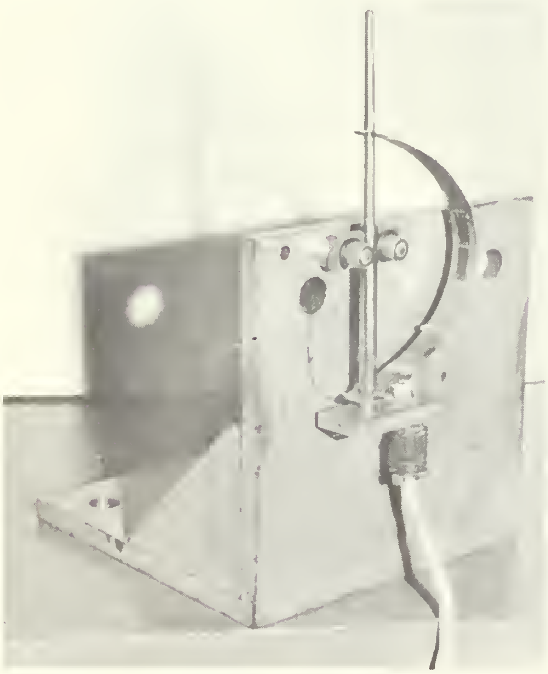


FIG. 1. PRIMARY TESTING EQUIPMENT

FIG. 2. TESTING MACHINE TRANSDUCERS

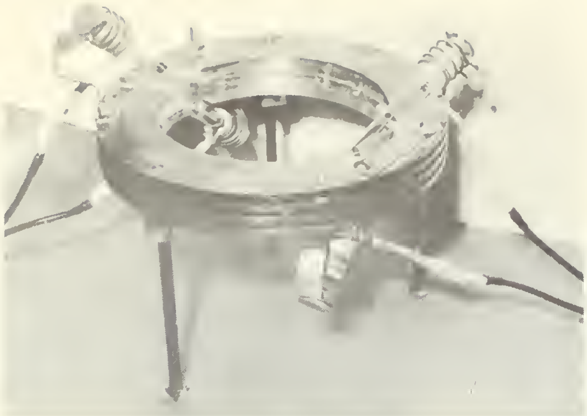
Micrometer Calibration
of "Positionometer"





Spring Beam
"Positionometer"

FIG. 3. CROSSHEAD MOVEMENT TRANSDUCER



Heating Apparatus

Ventilated Loading Head
and Chimney

Compressed Air Injection System

FIG. 4. HEATING AND VENTILATING SYSTEMS

FIG. 5. PREPARATION OF SPECIMENS FOR TEMPERATURE TESTING

The test cylinders to be tested in axial compression loading with combined heating were fitted with an additional thermocouple on the inside of the cylinder directly opposite one of the three outside thermocouples.

A one-half inch wide band for absorbing heat was prepared around the outside of all the cylinders tested with heating. This band was positioned with the outside thermocouples in the center of the band and was blacked with "Hydrograf" colloidal graphite. An aluminum reflector band was installed on each side of the heat band with glass insulating tape under the aluminum to prevent shorting of the thermocouples. The various stages of preparation are indicated in Fig. 5.

All of the specimens which were tested for ultimate strength at room temperature were instrumented with strain gages. The axial compression cylinders were instrumented with one pair of longitudinal gages and one pair of transverse gages. The hoop tension cylinders were fitted with one single transverse gage. The paired gages were attached with one gage inside and one gage outside.

IV. TEST PROCEDURES

Investigation conducted on preliminary specimens enabled the author to gain considerable experience with the testing equipment. These tests demonstrated that wrapping the cylinder ends with glass tape to prevent end failure when testing in axial compression was unsatisfactory because of the stress concentrations induced at the inside edge of the tape. The solution was potting the ends with epoxy as previously mentioned. Some of these samples were instrumented with six thermocouples for evaluation of various heating rates. The axial temperature differential within the 1/2 inch wide heated band was found to be less than $15^{\circ}F$. This led to the decision to place the thermocouple hot junctions in the center of the heat band for all other tests.

<u>Axial Compression Testing</u> - The axial compression specimens were compressed in the testing machine between the special ventilated loading head and a spherical seat. Prior to testing, the epoxy potted ends of each specimen were paralleled on a machine lathe using an expandable mandrel to grip the specimen from the inside.

Each pair of strain gages attached to the specimens to be tested at room temperature were wired into separate full-bridge circuits using gages installed on other specimens as dummy gages. These full-bridge circuits were then wired into the Sanborn recorder. For the room temperature tests, the load, crosshead movement, axial strain, and lateral strain were all recorded to failure.

For the temperature tests the cylinders were installed in the loading machine with the heating apparatus positioned as indicated in Fig. 6. The thermocouples were connected to the recorder and the "positionometer" was placed under the crosshead. Load was applied until a prescribed percentage of the room temperature ultimate load was reached. The heating and ventilating systems were then turned on and the load was maintained constant until failure occurred. The failure temperature was chosen as that temperature at which the specimen experienced yielding. Yielding was easily determined from the recorded data of the "positionometer". The load, crosshead movement, three outside thermocouple outputs, and one inside thermocouple output were recorded.

The calibration of the "positionometer" was confirmed at the end of each test.

Hoop Tension Testing - Hoop tension forces in the test specimens were obtained by installing the rubber plug shown in Fig. 7 in the center of the cylinder and then compressing it with the two steel pistons.

The strain gages of the specimens tested at room temperature were connected to dummy gages and wired into the recorder. The load, crosshead movement, and strain were recorded to failure.

The heating and ventilating systems were installed for the hoop tension temperature test in the same manner as for the axial compression temperature tests. Smoke removal was not noticeably affected by the increased distance between the ventilated loading head and the heating apparatus.

The specimen was then loaded to the prescribed stress level and the heating system was turned on. The load was maintained constant and was recorded with crosshead movement and temperatures to failure. The failure point was determined from the "positionometer" data. Here, again, the "positionometer" calibration was confirmed at the end of each test.

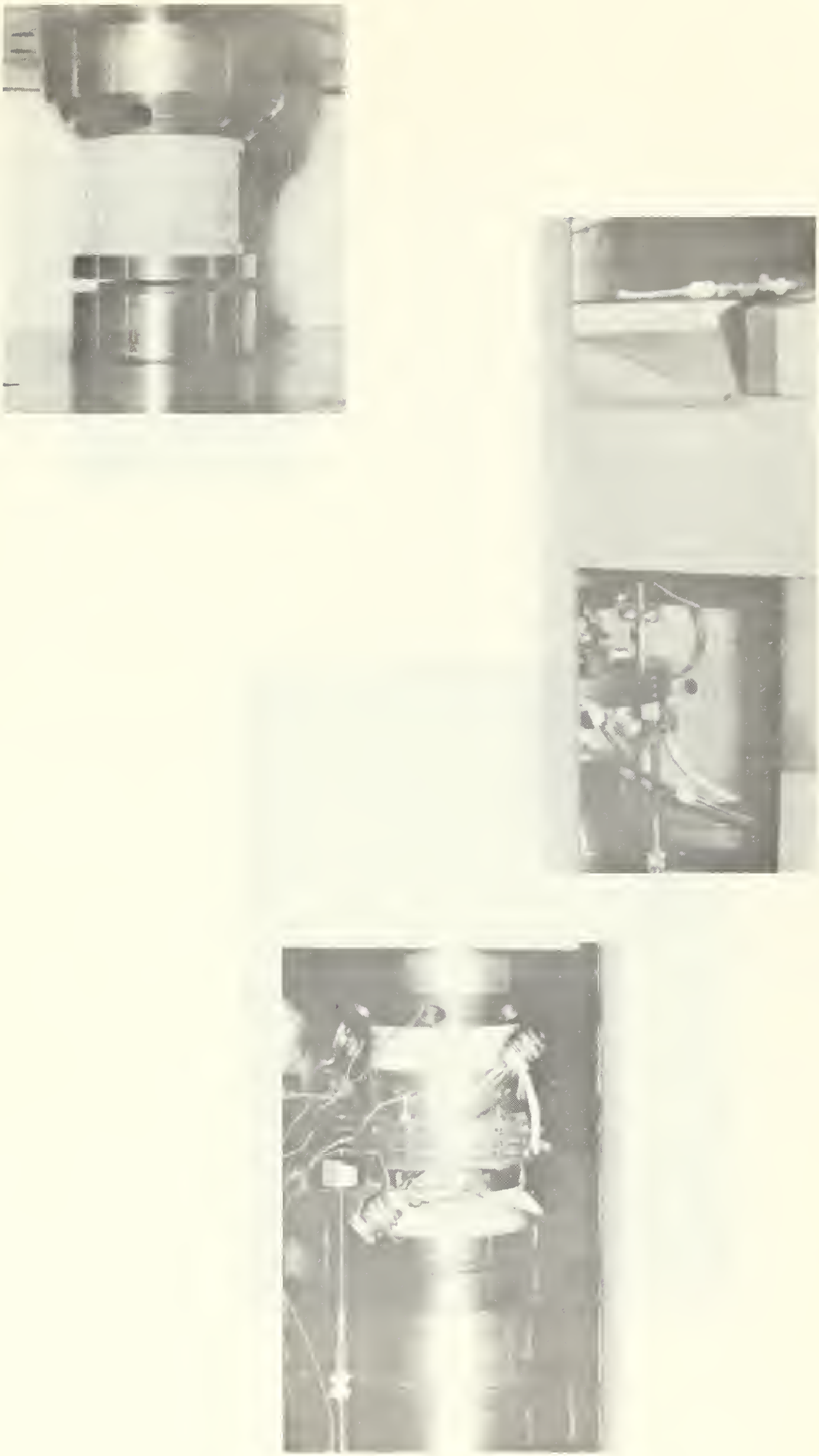


FIG. 6. AXIAL COMPRESSION TESTING

FIG. 7. HOOP TENSION TESTING

V. RESULTS

The results of the investigation are displayed in Fig. 8 through Fig.19. The primary data was obtained in the form of strip charts which are preserved on file and will not be presented. Data points are not indicated when the information was directly transcribed from the continuously recorded data.

Figure 8 shows the temperature-time profile for the outside and inside of the test cylinders produced by the heating device during this investigation. There was less than a $6^{o}F$ variation in the room temperature throughout the tests and less than a $30^{o}F$ variation in temperature between different tests for the same time of heating. The maximum radial temperature differential within the heated circle during any one test was approximately $25^{o}F$. For future testing a considerable variation in the heating rate can be obtained by an appropriate variation in the voltage supplied to the heaters.

Axial Compression Results - The ultimate stress versus percent inorganic binder is illustrated in Fig. 9. The absence of a steep slope for the almost straight line indicated that a considerable amount of inorganic binder may be present without greatly reducing the ultimate stress. The addition of 24% by weight of inorganic binder (from 1% to 25%) produced a corresponding reduction in ultimate stress of 28%. It is expected that as the percent inorganic binder exceeds 50% the curve would tend to flatten out and become almost horizontal at some low value of ultimate stress.

The axial compression Stress-Strain curves for the three values of inorganic binder content are illustrated in Fig. 10. It is interesting to note that the primary and secondary modulus phenomenon typical of epoxy matrix filament wound systems is clearly perceptible for the 1% and 10% system, but that the modulus of the 25% system is indicated by a continuous straight line. In accordance with standard practice, the secondary modulus is reported for the 1% and 10% systems. The values of local strain measured by the strain gages were always within 12% of the average strain values. The values of strain indicated in Fig. 10 are average strains obtained from the "positionometer". These results could not have been found from the strain gages used since they separated from the specimens at strain levels of approximately 0.4%. Rubber strain gages could have been used; however, their large size would have made

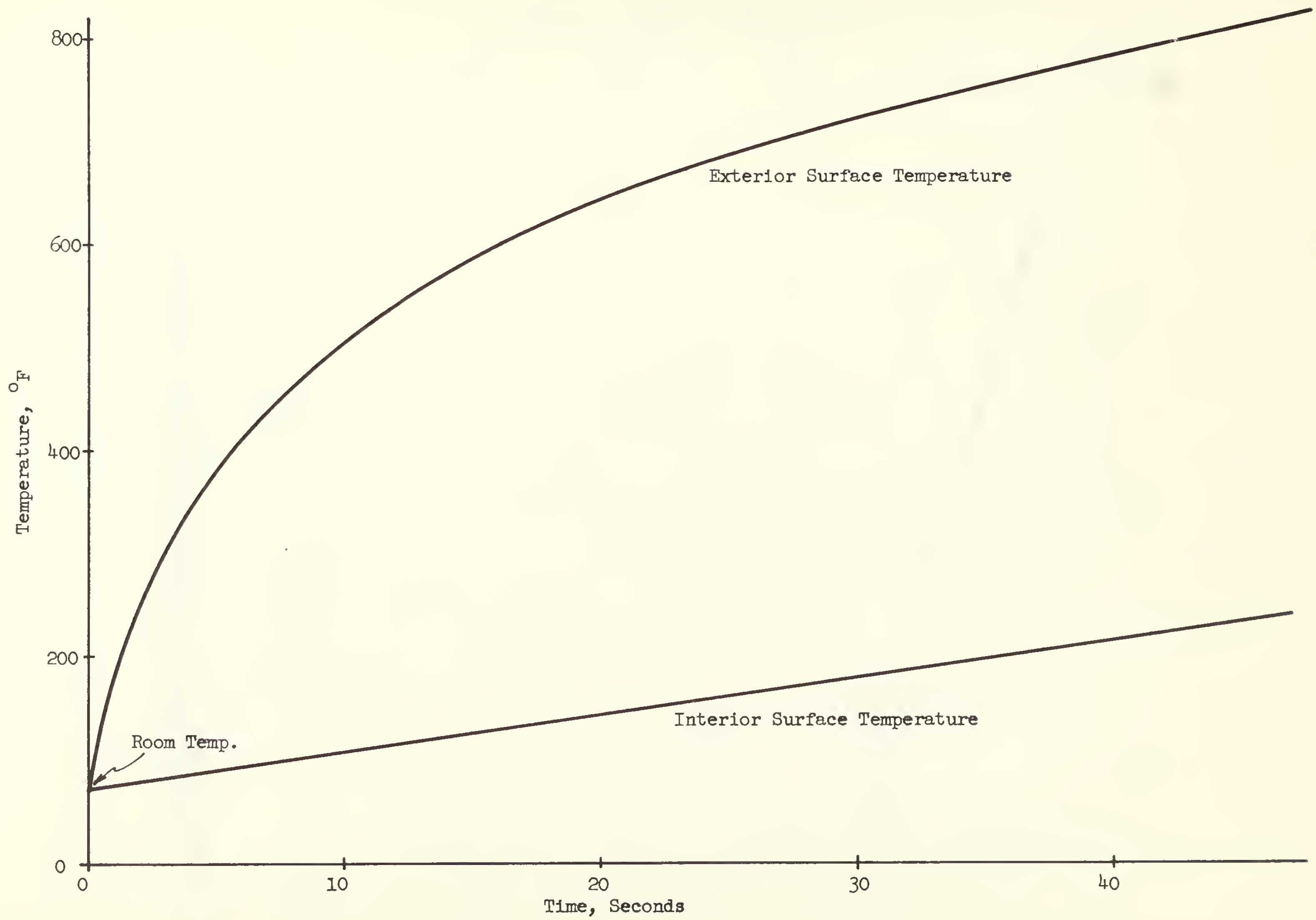


FIG. 8. CYLINDER WALL TEMPERATURE VERSUS TIME FOR HEATING WITH EXTERNAL CIRCULAR HEATER

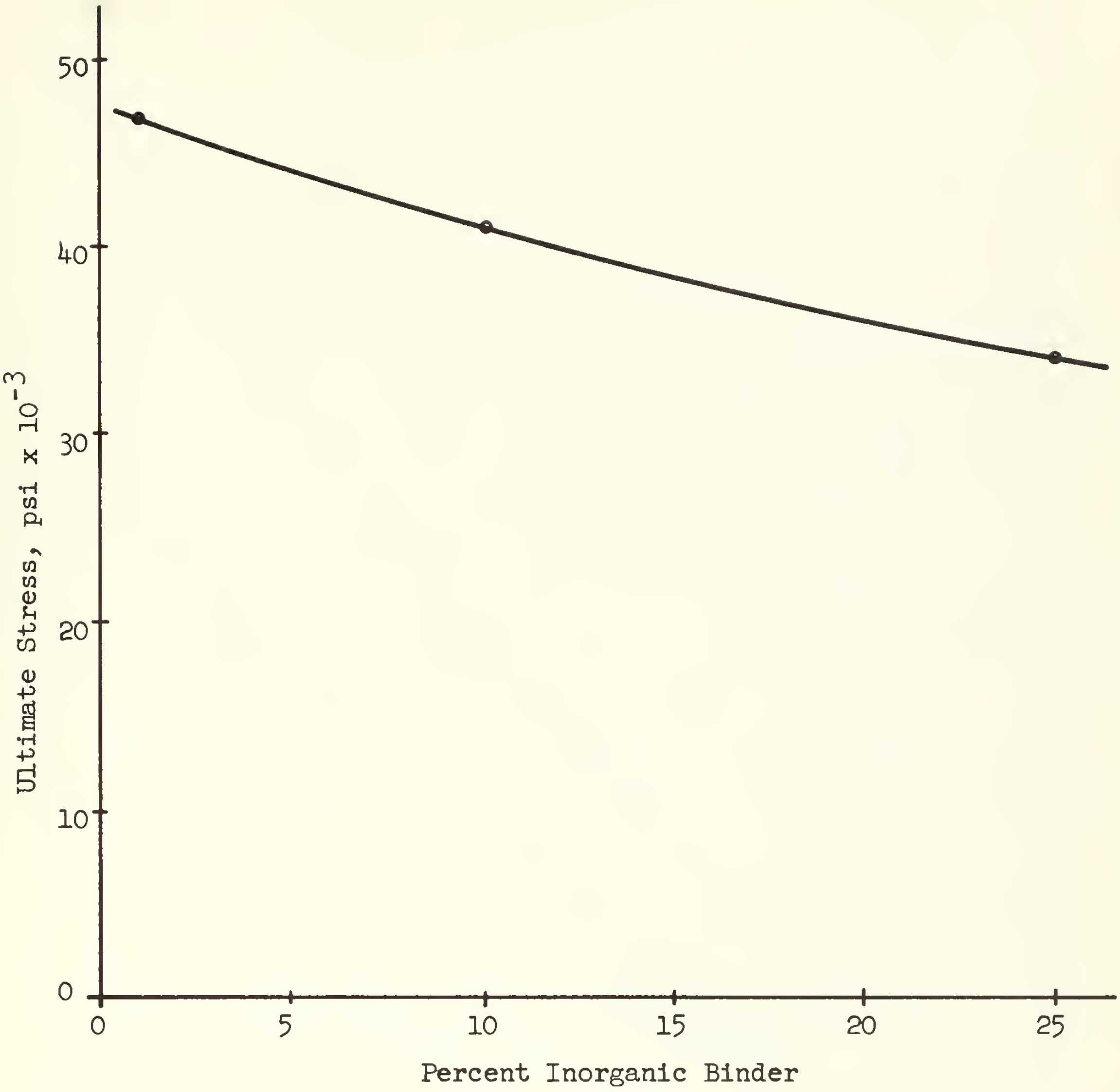


FIG. 9. ULTIMATE STRESS FOR AXIAL COMPRESSION VERSUS PERCENT INORGANIC BINDER, BY WEIGHT

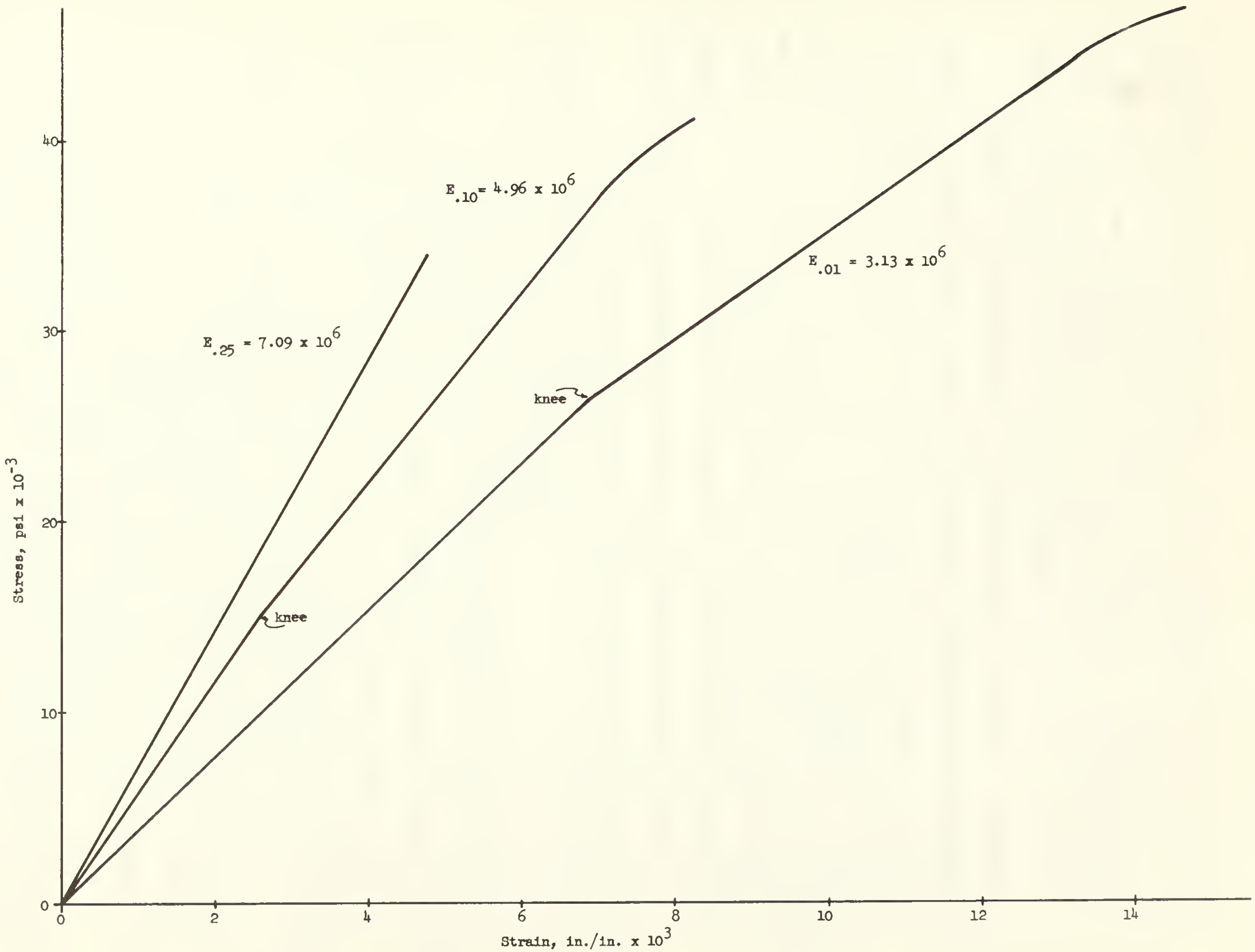


FIG. 10. MODULUS OF ELASTICITY FOR AXIAL COMPRESSION OF INORGANIC BONDED FILAMENT WOUND CYLINDERS

them difficult to use for these tests. The "positionometer" used with the Sanborn recorder offers the advantage of being able to easily record the average strain of the specimens for all the tests, including the elevated temperature tests. When investigating the usual performance of a particular specimen, this additional information could be quite valuable.

Figure 11 illustrates the axial compression modulus of elasticity versus percent inorganic binder content. This curve should become horizontal at some value of modulus less than 10×10^6 psi as the percent inorganic binder is increased above the 25% level. The substantial increase in modulus with an increase in inorganic binder content indicates the desirability of an inorganic bonded system for thin shell structures such as missiles where the modulus as a function of the critical buckling stress is quite often the major structural consideration. For the system investigated, a 126% increase in modulus was obtained with a corresponding decrease in ultimate stress of 28%.

Poisson's ratio for each percent binder content was determined from the recorded strain gage data and is illustrated in Fig. 12. The values shown were determined at 50% of the ultimate stress.

The results of the axial compression heating tests are displayed in Fig. 13. The room temperature ultimate stress forms the upper stress limit of these curves while the lower limit is that value of stress which can be maintained by the inorganic bonded structure without the epoxy matrix.

The behavior of the 1% system is as expected since it has virtually no strength without the epoxy matrix. This curve rapidly approaches a vertical line as the temperature exceeds the yield temperature of the epoxy.

The 10% system indicates a greater compressive strength than the 1% system as the epoxy yield temperature is approached. However, this data does not indicate any gain of compressive strength at elevated temperatures of the 25% system over the 10% system except at low stress levels.

The temperatures indicated in Fig. 13 are for the outside surface only. If the specimens had been heated to a uniform temperature prior to loading, the only meaningful results would be at temperatures below the epoxy yield temperature. This would leave very little for discussion since there were no significant changes in structural properties due to heating until the epoxy yield temperature was approached.

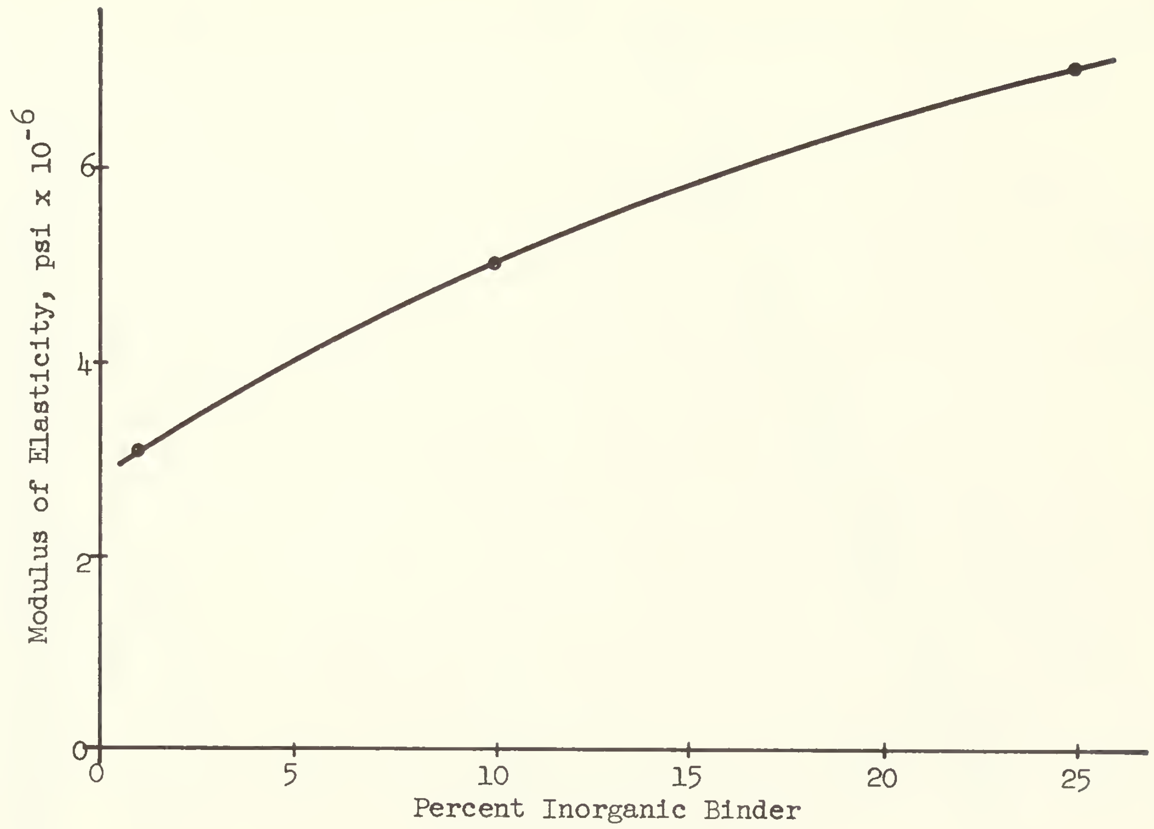


FIG. 11. MODULUS OF ELASTICITY FOR AXIAL COMPRESSION VERSUS PERCENT INORGANIC BINDER, BY WEIGHT

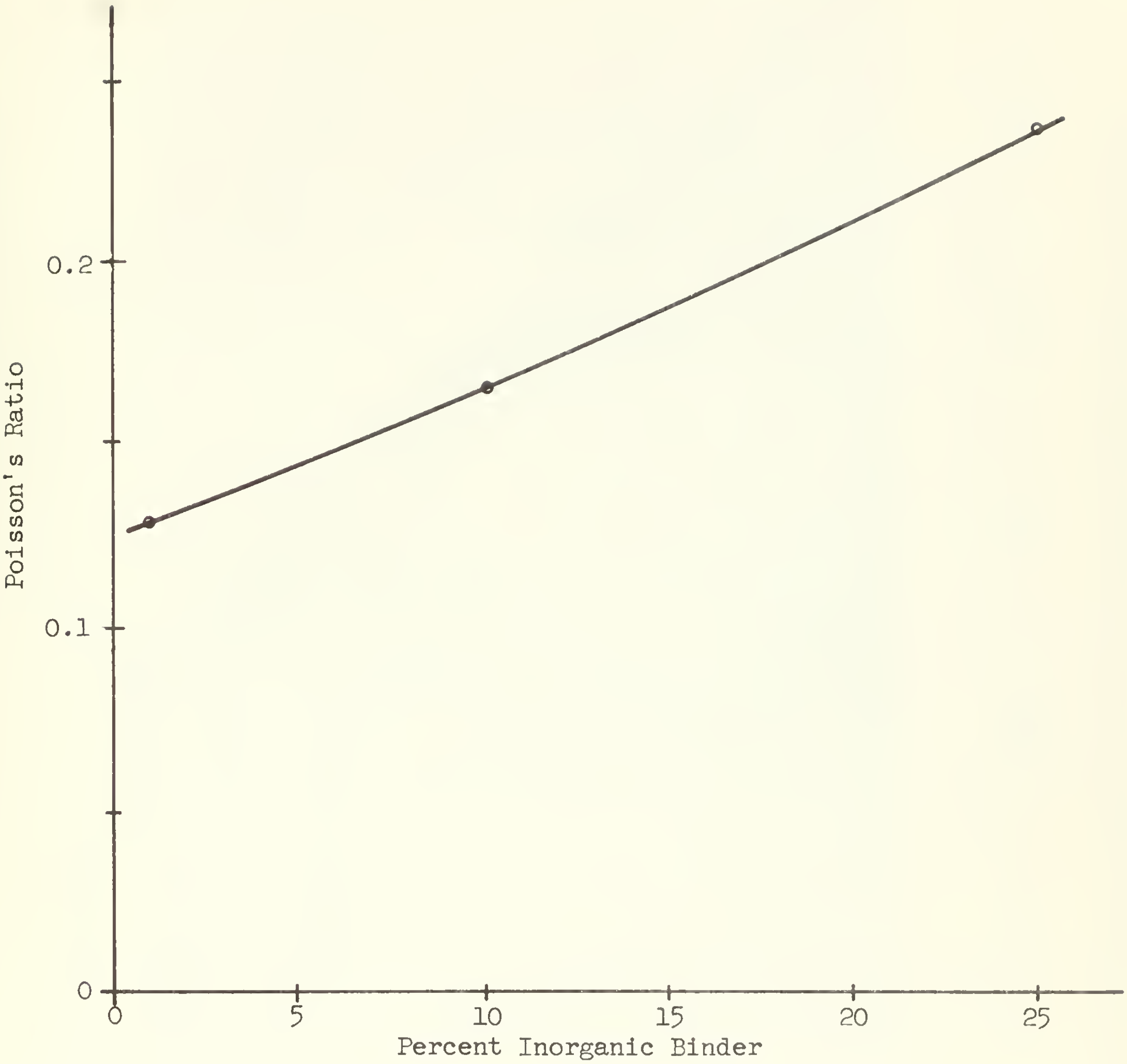


FIG. 12. POISSON'S RATIO FOR AXIAL COMPRESSION VERSUS PERCENT INORGANIC BINDER, BY WEIGHT

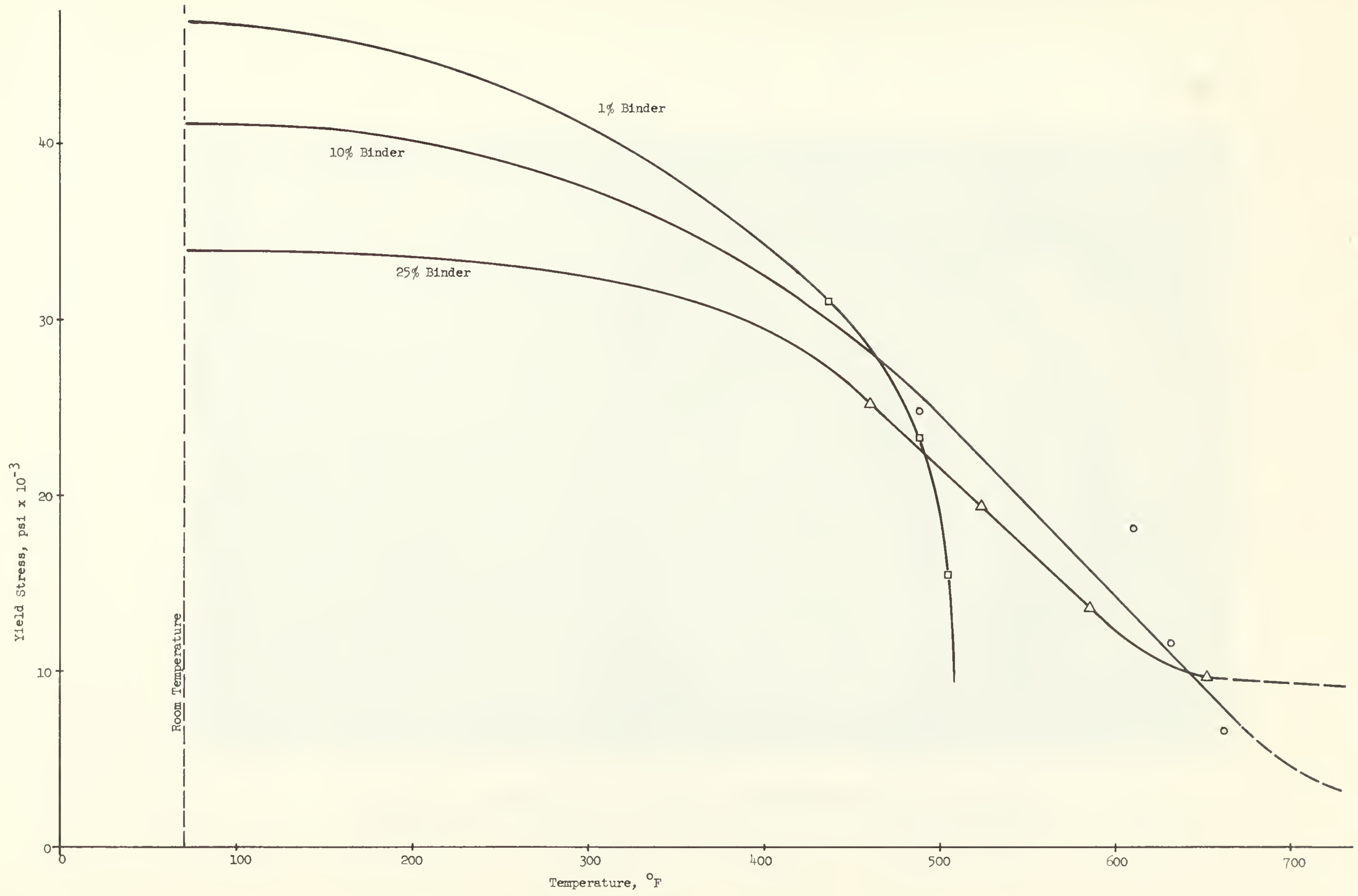


FIG. 13. AXIAL COMPRESSION YIELD STRESS VERSUS TEMPERATURE FOR INORGANIC BONDED FILAMENT WOUND CYLINDERS

FIG. 14. FAILURE OF AXIAL COMPRESSION SPECIMENS

Figure 14 shows the types of test specimen failure which occurred for axial compression testing at room temperature and at elevated temperatures. There was no significant difference between the failure of specimens with different binder densities. The room temperature failure was always a fracture and the elevated temperature failure was always failure due to yielding.

<u>Hoop Tension Results</u> - The data obtained from testing the original hoop tension specimens was eliminated as a possible indicator of inorganic binder effects on structural properties due to processing errors. One such error was a difference in angle of wind between the various specimens of approximately 2^{o}. This represents an angle of wind variation of 8% which was sufficient to produce erratic test results.

It was decided to produce one more master cylinder from which specimens of different inorganic binder content would be produced. This insured constant angle of wind but did not allow enough specimens for temperature testing. Specimens were made from this last master cylinder which contained 2%, 10%, and 24% inorganic binder content. The processing was exactly the same as for the earlier specimens except that thermocouples were not installed. These new specimens were instrumented with strain gages and tested at room temperature using the previously described hoop tension testing technique. The results of these tests are illustrated in Fig. 15 through Fig. 17.

The ultimate stress versus percent inorganic binder is illustrated in Fig. 15. The reduction in ultimate stress corresponding to an increase in binder content is seen to be greater for hoop tension than for axial compression. However, this figure indicates that a reasonable amount of inorganic binder may be present without substantially reducing the ultimate stress.

The hoop tension Stress-Strain curves for the three values of inorganic binder content are illustrated in Fig. 16. There is no evidence of the primary and secondary modulus phenomenon. The relative change in modulus corresponding to a change in the inorganic binder content indicates that there would be little to gain in terms of modulus increase by increasing the inorganic binder content above the 10% level. This is further illustrated in the modulus of elasticity versus percent inorganic binder curve of Fig. 17

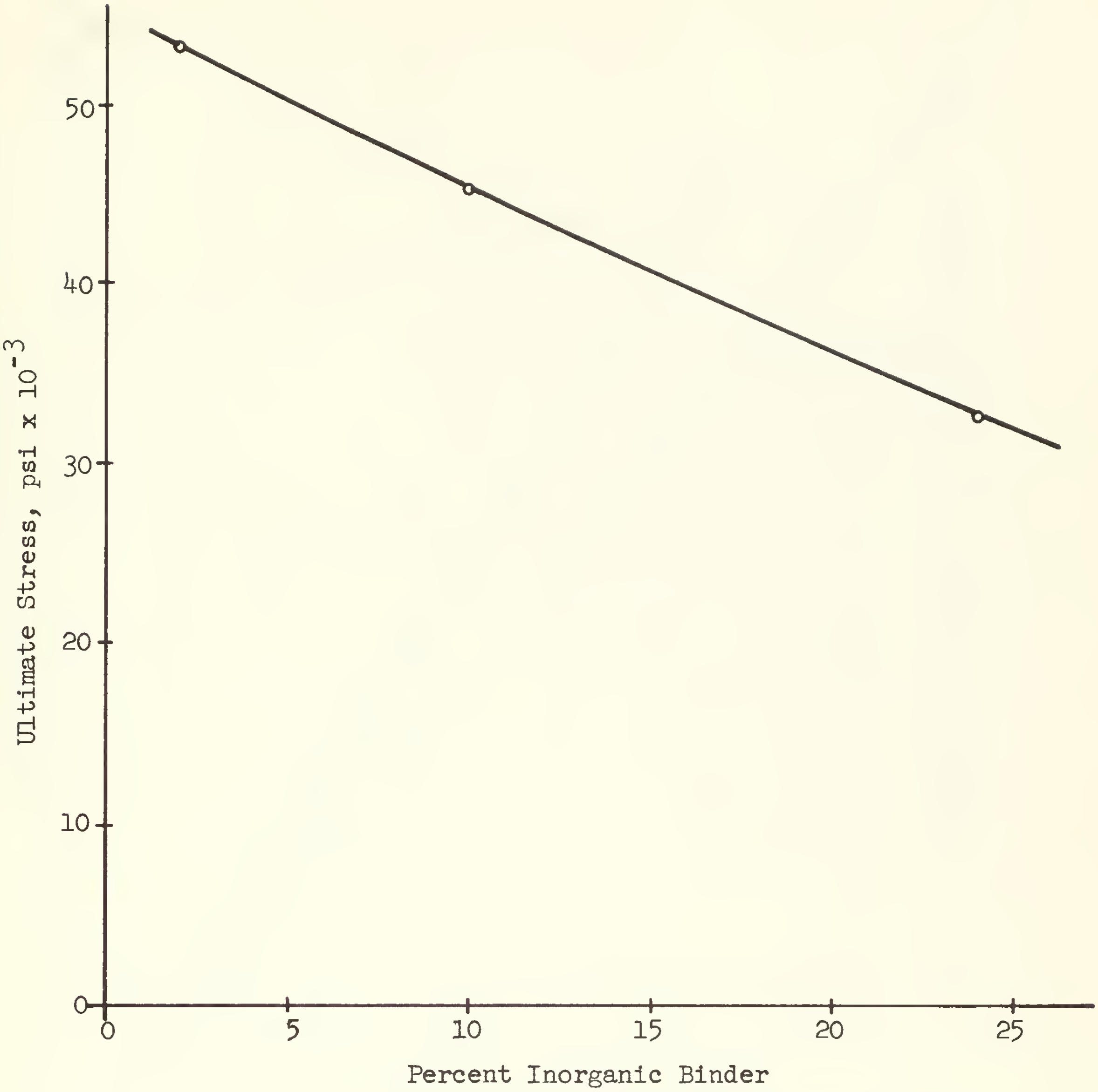


FIG. 15. ULTIMATE STRESS FOR HOOP TENSION VERSUS PERCENT INORGANIC BINDER, BY WEIGHT

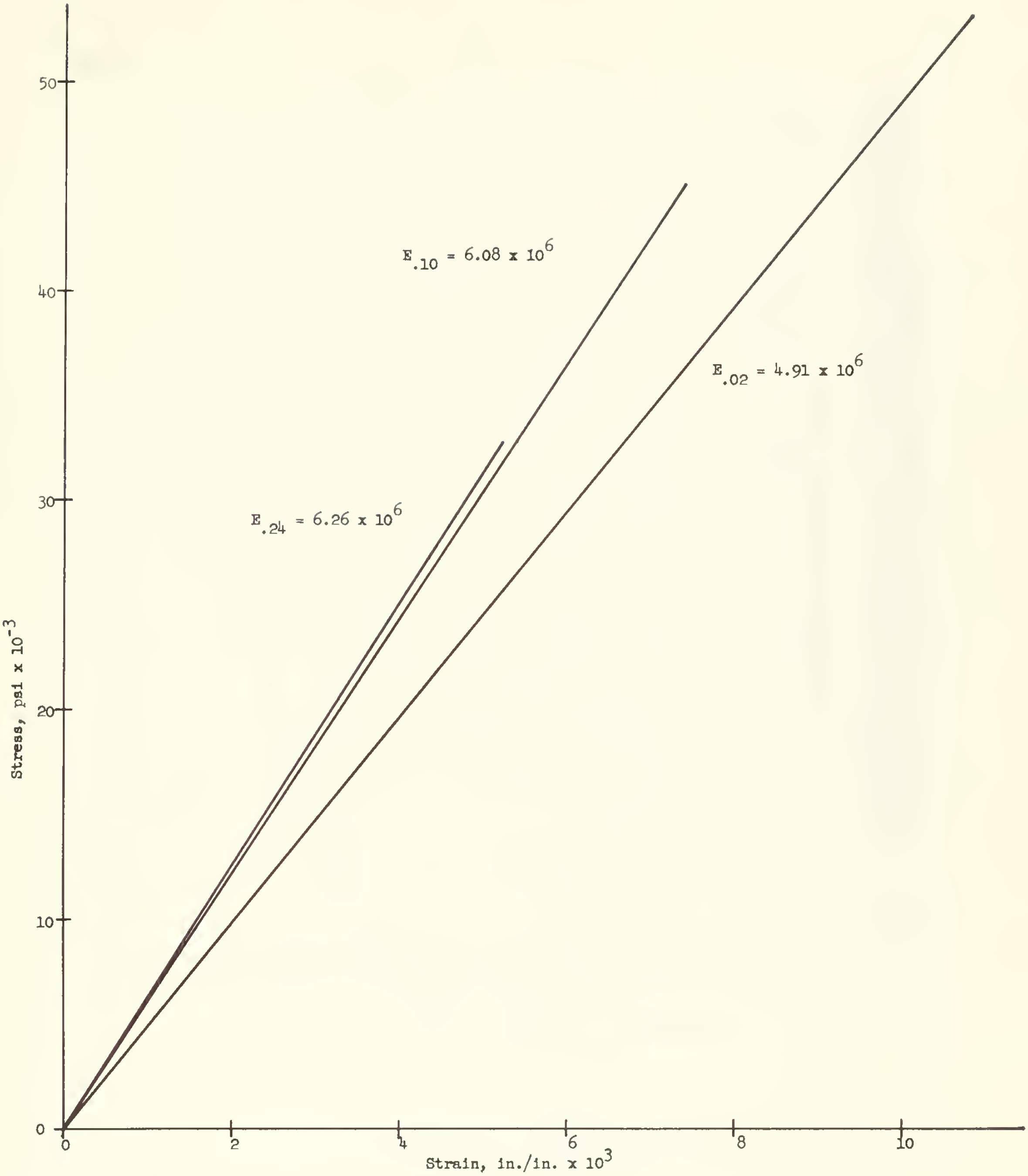


FIG. 16. MODULUS OF ELASTICITY FOR HOOP TENSION OF INORGANIC BONDED FILAMENT WOUND CYLINDERS

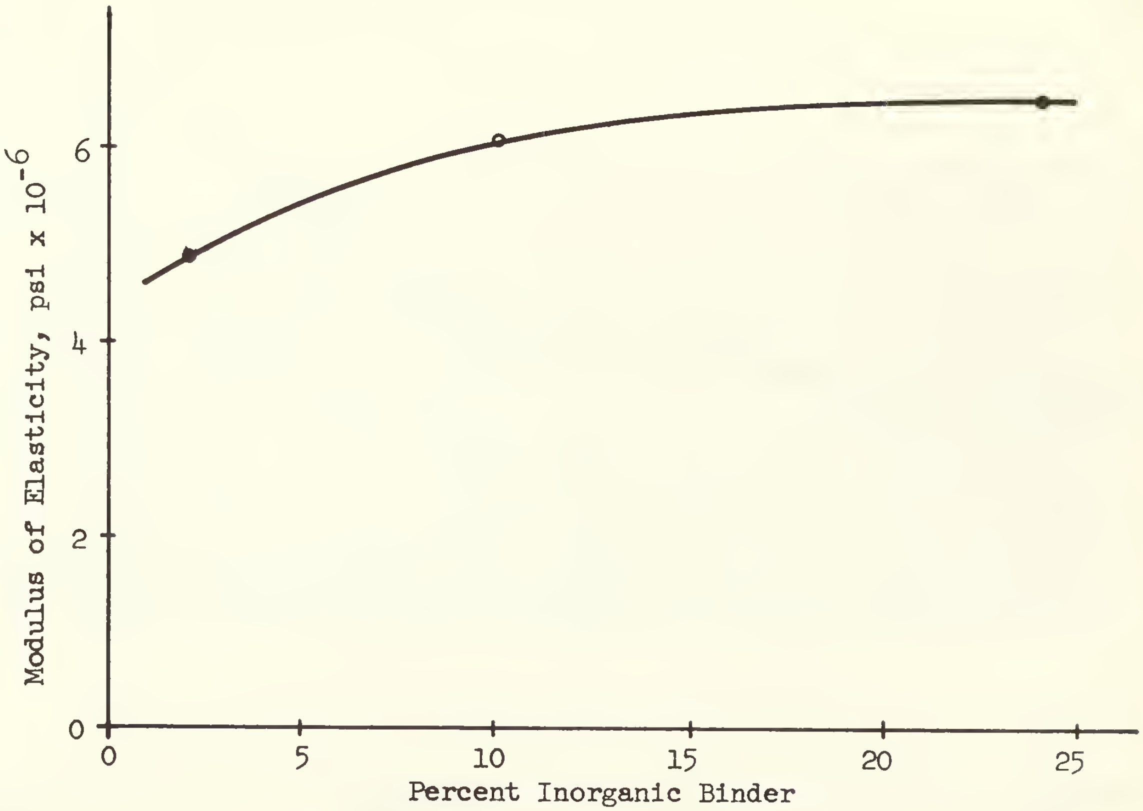


FIG. 17. MODULUS OF ELASTICITY FOR HOOP TENSION VERSUS PERCENT INORGANIC BINDER, BY WEIGHT

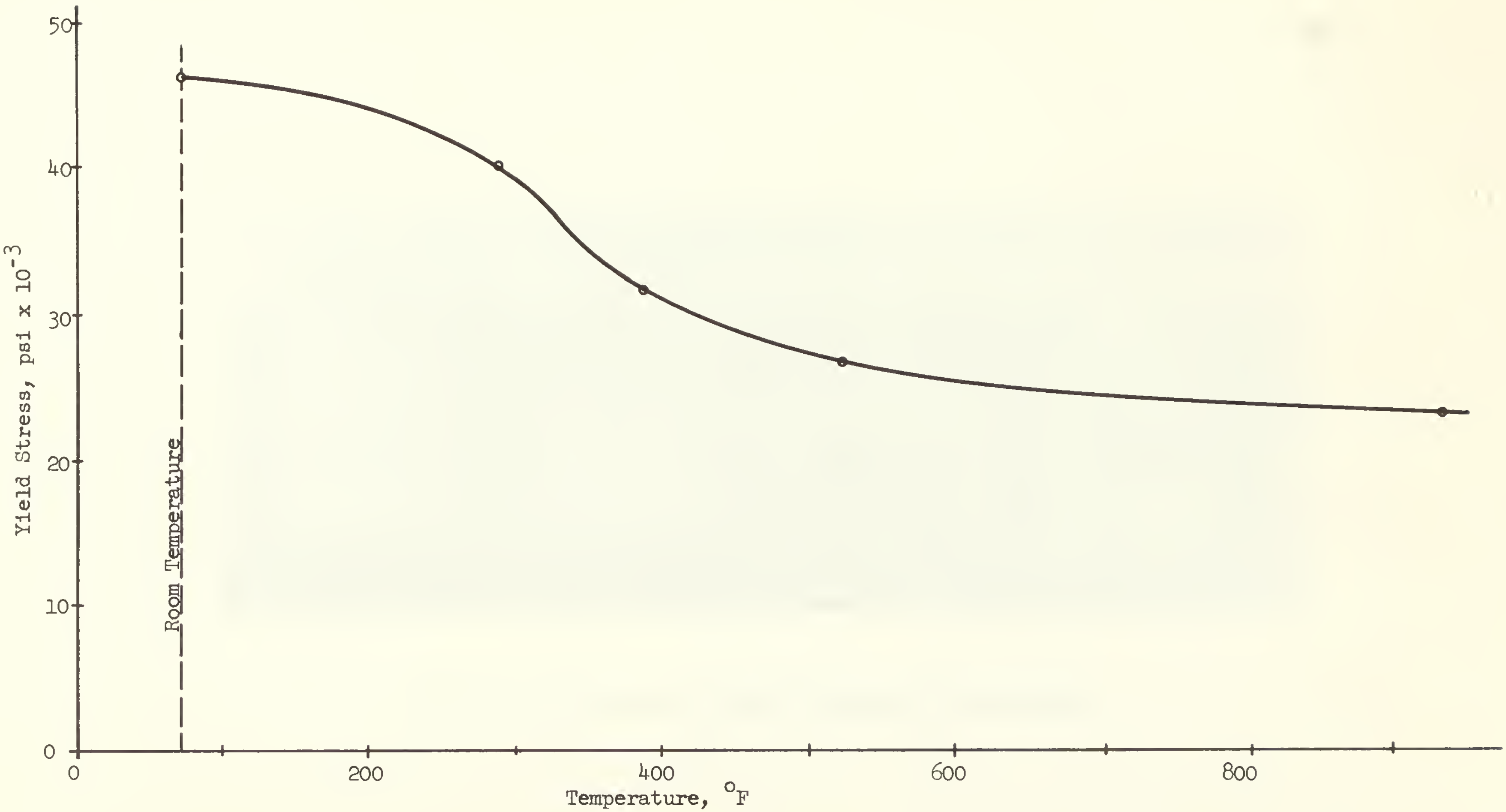


FIG. 18. HOOP TENSION YIELD STRESS VERSUS TEMPERATURE FOR INORGANIC BONDED FILAMENT WOUND CYLINDERS WITH 10% BINDER CONTENT

FIG. 19. FAILURE OF HOOP TENSION SPECIMENS

The hoop tension yield stress versus temperature results for the original 10% binder specimens are displayed in Fig. 18. These particular specimens were free from processing errors and their test results are useful in illustrating a typical stress-temperature reaction. Unfortunately, the temperature results of the 1% and 25% systems were too erratic to be presented for comparison. The results of the 1% system did indicate a lack of any substantial decrease in ultimate stress with an increase in temperature. For the particular case of a 26° helical wound cylinder there is evidence that the lower binder content cylinders will retain higher ultimate hoop tension stresses throughout the heating range. Such a conclusion can also be arrived at intuitively since there would be no noticeable loss in ultimate stress as the temperature of a specimen in pure tension passed through the yield temperature of the matrix material.

Typical hoop tension specimen failure is shown in Fig. 19. Both the room temperature specimens and the elevated temperature specimens exhibited fracture type failure. The lower density specimens tended to fail along lines parallel to the angle of wrap of the yarn while the higher density specimens failed parallel to the axis of rotation.

VI. CONCLUSIONS AND RECOMMENDATIONS

At room temperature there is no indication of a drastic reduction in either axial compression ultimate stress or hoop tension ultimate stress as the inorganic binder content is increased to levels of approximately 25%. At temperatures above the matrix yield temperature there is a definite increase in axial compression ultimate stress with the addition of inorganic binder. The hoop tension ultimate stress at elevated temperatures does not increase as inorganic binder is added to specimens with a 26° helical angle of wind.

For axial compression and hoop tension there is a significant increase in the modulus of elasticity associated with an increase in inorganic binder content up to 10%. Above 10% binder content the increase in hoop tension modulus is relatively small. As the inorganic binder content is increased there is a corresponding increase in the axial compression Poisson's ratio.

The primary and secondary modulus phenomenon typical of epoxy matrix filament wound systems was perceptible for cylinders with as much as 10% inorganic binder content when tested in axial compression. This phenomenon was not detectable for the hoop tension modulus.

There is some question as to what extent the deleterious effects of moisture on 'quartz" fiber were manifested in the processing of the test specimens. Specimens are presently being manufacture using "S" glass which does not present any problems when processed with aqueous solutions of binder. Inorganic binders other than silica are also being evaluated.

In the development of the "Lock-Heat" structure, many samples were manufactured with various yarns, binders, and matrix materials. The area of primary interest was thermal properties and very little attention was given to the structural properties of the specimens. All of the specimens presently being produced are tested for hoop tension ultimate stress. However, this furnishes only a small portion of the data necessary for proper structural design. There is no doubt that the specimens presently being produced could yield considerably more structural information with a relatively small additional investment in equipment.

The results indicate the possibility of designing a plastic-filled inorganic bonded structure capable of serving as the primary structure and also the heat shielding of a supersonic vehicle. However, if any serious interest in this possibility is to persist, an organized program for structural testing must be initiated.

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